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# U-shape panda polarization-maintaining microfiber sensor coated with graphene oxide for relative humidity measurement

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***Abstract*—A new U-shape panda polarization-maintaining fiber (PPMF) based microfiber interferometer coated with graphene oxide (GO) film was proposed and experimentally demonstrated for relative humidity (RH) sensing. Experimental results show that the U-shape sensor has refractive index (RI) sensitivity of 1692.5 nm/RIU in the RI range of 1.33 when the diameter of the taper waist is 10.08 μm. The surface of the U-shape sensor was then modified chemically and coated with a thin layer of GO film (59.64 nm) for RH detection and the sensitivity is proportional to RH: as RH increases from 30% to 98%, the sensitivity increases from 0.111 to 0.361 nm/%RH and the response time is 0.28 s. In addition, the cross sensitivity to temperature, stability, reproducibility, and response/recovery time of the RH sensor were studied in detail. The proposed U-shape fiber RH sensor has advantage of high sensitivity, good reproducibility and fast response (0.28 s), which has potential application in areas requiring dynamic measurement of RH variations such as industrial product fabrication process control and breath state monitoring.**

***Index Terms*—Relative humidity; microfiber interferometer; U-shape fiber; graphene oxide**

## I. INTRODUCTION

IN the last ten years, different types of optical fiber relative humidity (RH) sensor structures were reported, such as Fabry-Perot interferometer [1], Mach-Zehnder interferometer (MZI) [2-3], Michelson interferometer [4], side-polished fibers [5], long period grating [6], fiber bragg grating [7], surface plasmon resonance [8] etc. In general, the fiber sensor can be classified by two types: with and without coating RH sensitive material. For the sensor without coating RH sensitive material, Shao *et al*. proposed a Michelson RH sensor based on photonic crystal fiber with the sensitivity of -0.166 dB/%RH in the RH range of 30-90 %RH [9]. Liu *et al*. reported a tapered small core single mode fiber sensor for RH sensing with a maximum RH sensitivity of 183 pm/%RH in the RH range of 90.4-94.5 %RH [10]. Sun *et al*. proposed a high-birefringence microfiber based Sagnac interferometer RH sensor, which has a maximum RH sensitivity of 201.25 pm/%RH in the RH range 30-90 %RH [11]. To improve RH sensitivity, most commonly the surface of the optical fiber sensor is coated with RH sensitive materials, such as polyvinyl alcohol (PVA), agarose, graphene oxide (GO), zinc oxide (ZnO), chitosan, Polyethylene oxide (PEO), and polycarbonate. Chen *et al*. proposed a microfiber interferometry coated with PVA for RH sensing and the RH sensitivity is 0.119 nm/%RH [12]. Mallik *et al*. proposed a microsphere resonant cavity sensor structure coated with a thin layer of agarose-coated silica for RH detection, which has maximum sensitivity of 518 pm/%RH in the RH range from 30% to 70% [13]. Gao *et al*. reported a new RH sensor structure by coating a thin layer of rGO on the surface of a hollow core optical fiber with an inner diameter of 70 μm and the RH sensitivity is 0.22 dB/%RH [14]. Lokman *et al*. deposited ZnO nanowires on the surface of the dumbbell-shaped fiber Mach-Zehnder sensor for RH detection within a range of 35%-60%RH and the total wavelength shift is 0.490 nm [15]. Chen *et al*. proposed a RH optical fiber sensor based on the Fabry-Perot interferometer and coated water-sensitive natural polymer chitosan on its surface, which has RH sensitivity of 0.13 nm/%RH and response time of 380 ms in the RH range of 20%-95% [16]. Wu *et al*. proposed a singlemode-small core-singlemode optical fiber structure and deposited moisture sensitive material of PEO onto the small-core optical fiber for RH sensing. The PEO coated fiber RH sensor can achieve a maximum sensitivity 4.30 nm/%RH and 0.50 nm/%RH ranging from 80% to 83%RH and from 83% to 95% RH respectively [17]. Woyessa coated polycarbonate on the surface of fiber Bragg grating and the RH sensitivity is only 7.3 pm/%RH [18].

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GO, a common derivative of graphene, is widely used in the field of RH sensing due to its excellent dispersibility, hydrophilicity, larger aspect ratio and fast reaction with water molecules, enabling it as an excellent RH sensitive material for various RH sensing devices [19-22]. GO contains two *sp2*- and *sp3*- hybrid carbon atoms. There are abundant oxygen-containing functional groups, such as hydroxyl, epoxy, and carboxyl groups on the base plane and the edge of the sheet. Because of these unique structural advantages, GO can be modified to the surface of optical fiber sensors by chemical bonding method, which has been widely used in the field of biosensing [23, 24]. Deng *et al*. proposed a GO film coated Mach-Zehnder interferometry for measuring RH with RH sensitivity 0.263 dB/RH% over RH range 35% to 85% [25]. Liu *et al*. proposed to functionalize a thin layer of GO nanosheets (49.2 nm) on a specific designed long-period grating for ultra-sensitive specific binding detection between antibody and antigen [26]. In addition, GO also has been applied in ultrahigh resolution thickness measurement and mode locked lasers [27, 28].

This paper proposed and investigated a tapered panda polarization-maintaining fiber (PPMF) based U-shape microfiber sensor. The sensor was coated with GO film using chemical binding method with the amino group of APTES ((3-Aminopropyl)triethoxysilane, purchased from Aladdin) [29]. The influence of GO film thickness and waist diameter on the RH sensitivity was experimentally studied and analyzed, and the response time, temperature sensitivity, stability and reproducibility were investigated in detail.

## II. Experimental Investigation

### A. Fabrication of U-shape PPMF microfiber sensor

To fabricate a U-shape PPMF microfiber sensor, a spherical shape was made by arc discharge at the end of a single-mode fiber (SMF, G.652D) using a fusion splicer (Fujikura 80 C). The arc discharge intensity and duration time are 100 bit and 2000 ms, respectively. A short section PPMF (15 mm) was sandwiched between two SMFs with spherical connections. The PPMF part is then tapered to few diameters by an optical fiber tapering system (OC-2010, JILONG). The fiber taper with reduced diameter has strong evanescent wave, which will interact with surrounding materials and thus improve sensor sensitivity significantly [30]. The tapered PPMF part is then bent into a U-shape sensor structure as illustrated in Fig. 1 (a). A schematic diagram of the cross-section of the PPMF is shown in Fig. 1 (b). As light was injected from the input SMF to the PPMF, due to the spherical part of the structure, both core and cladding modes excited by the spherical part will propagate along the PPMF, which will interference and be collected to the output SMF core by the second spherical part, resulting in transmission peak and dip change at the output the sensor [31, 32]. Figure 1(b) shows simulated result of how core and cladding modes were excited and propagated as light (1550 nm) incident from SMF to the spherical part and PPMF.

The scanning electron microscope (collected by FESEM, FEI Nano SEM450*) images of the spherical part and bend section are illustrated in Fig. 1 (c) and (d).

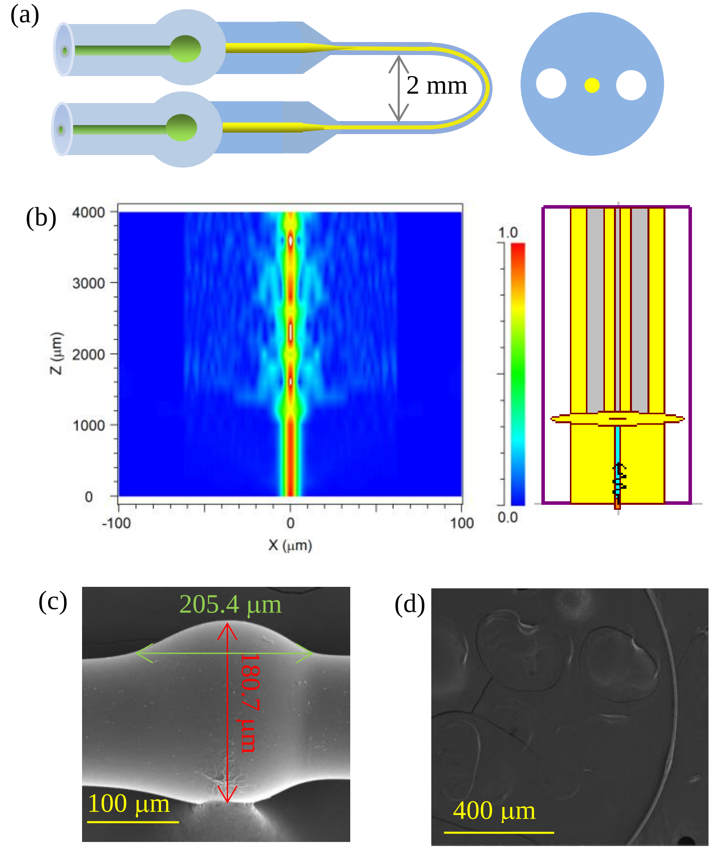
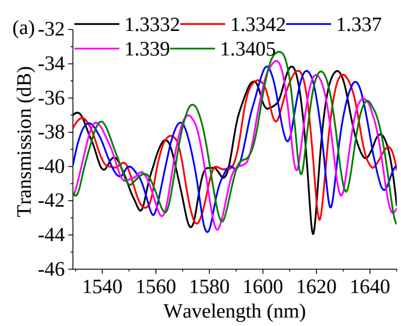



Fig. 1. A schematic diagram of (a) U-shape microfiber structure and the cross-section of PPMF; (b) simulated light transmission from SMF to PMOF through the spherical session and simulation model; SEM image of (c) spherical part; (d) U-shape part

### B. RI sensing characterization

The RI sensitivity of the proposed U-shape microfiber sensors with different taper waist diameters is analyzed. Figure 2(a) shows the spectral response of a U-shape microsensor with a taper waist diameter of 10.08 µm immersed into different surrounding liquids with RI ranging from 1.3332 to 1.3405. As shown in Fig. 2(a), as RI increases, the wavelength of the sensor shifts toward longer wavelength monotonically. Figure 2(b) summarized the wavelength shift vs. RI with different taper waist diameters, showing that the RI sensitivity increases from 752.2 nm/RIU to 1692.5 nm/RIU as the diameter of the waist taper decreases from 26.28 to 10.08 µm in the RI range from 1.33 to 1.34. The smaller the taper waist diameter, the higher the sensitivity. The sensor with a taper diameter of 10.08 µm has the sensitivity of 3079.96 and 9465.8 nm/RIU in the RI range from 1.37 to 1.38 and from 1.40 to 1.41 respectively as shown in Fig. 2 (c, d), indicating that as the surrounding RI is closer to that of the fiber, the sensor has higher RI sensitivity.



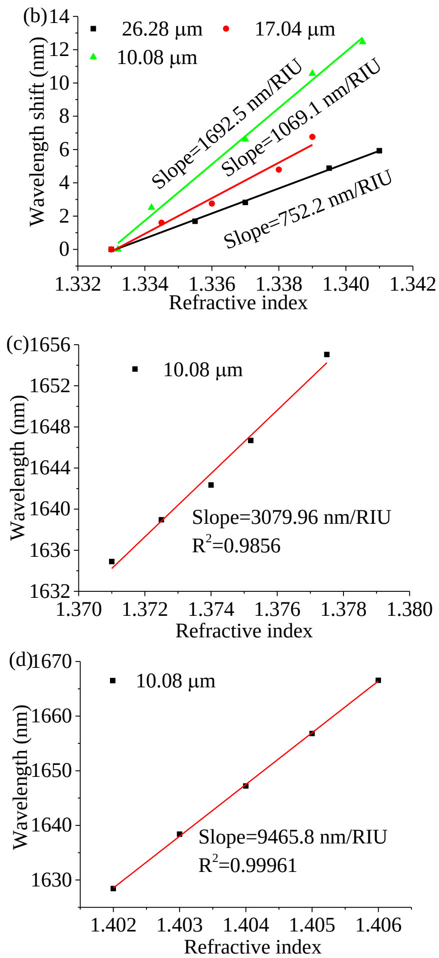



Fig. 2. (a) Measured spectral response of a U-shape microfiber sensor with a taper waist diameter of 10.08 μm in the RI range of 1.33; the RI sensitivities of sensors (b) with different taper waist diameters in the RI from 1.33 to 1.34; with a taper waist diameter of 10.08 μm in the RI range of (c) 1.37 to 1.38 and (d) 1.40 to 1.41.

### *C. Microfiber sensor functionalization and RH sensing mechanism*

The GO film, a collection of micro-crystals stacked on top of each other, has strong ability to absorb water vapor [33]. As a result, a thin layer of GO film is functionalized on the PPMF microfiber sensor to enhance the RH sensitivity. The mechanism of the binding process between GO film and water molecules is illustrated in Fig. 3. To improve the adhesion of GO film to the microfiber sensor surface, a chemical bond modification method to deposit GO (purchased from Soochow Hengqiu Graphene Technology Co., Ltd.) on the U-shape fiber sensor was used in the experiment.

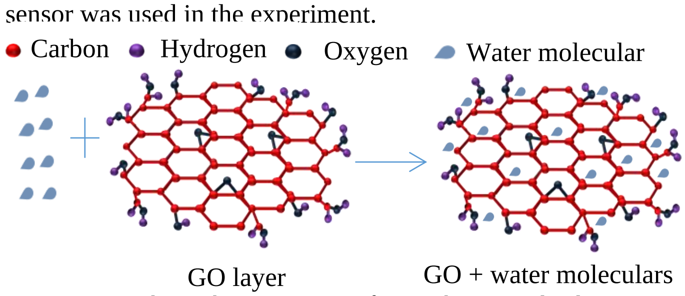



Fig. 3. The combination process of GO with water molecules.

The detailed functionalization steps are illustrated as follows:

1) The U-shape sensor is firstly cleaned with ethanol and then rinsed with deionized water three times to remove surface impurities and pollutants.

2) The cleaned U-shape sensor is then immersed in 1 mol/mL KOH solution for 1 hour to produce abundant silanol groups (Si-OH) on microfiber surface (Fig. 4(a)). The sensor was then rinsed with ethanol and deionized water in sequence for 3 times.

3) The above treated microfiber sensor is immersed into a 5% (V/V) APTES ethanol solution (freshly prepared) for 1 hour, which can react with the hydroxyl group to form the Si-O-Si chemical bond (Fig. 4(b)) and then rinsed with ethanol and deionized water in sequence. At last, the U-shape sensor is placed inside an oven at 45 °C to dry the APTES molecular layer for half an hour.

4) The above treated U-shape microfiber sensor is immersed in the GO solution and then lifted at a rate of 0.25 mm/s. The epoxy groups of GO react with the amino groups of APTES-silanized fibers. Finally, the U-shape sensor is placed inside an oven at 40 °C for 4 hours and formed the uniform GO film (Fig. 4 (c)).

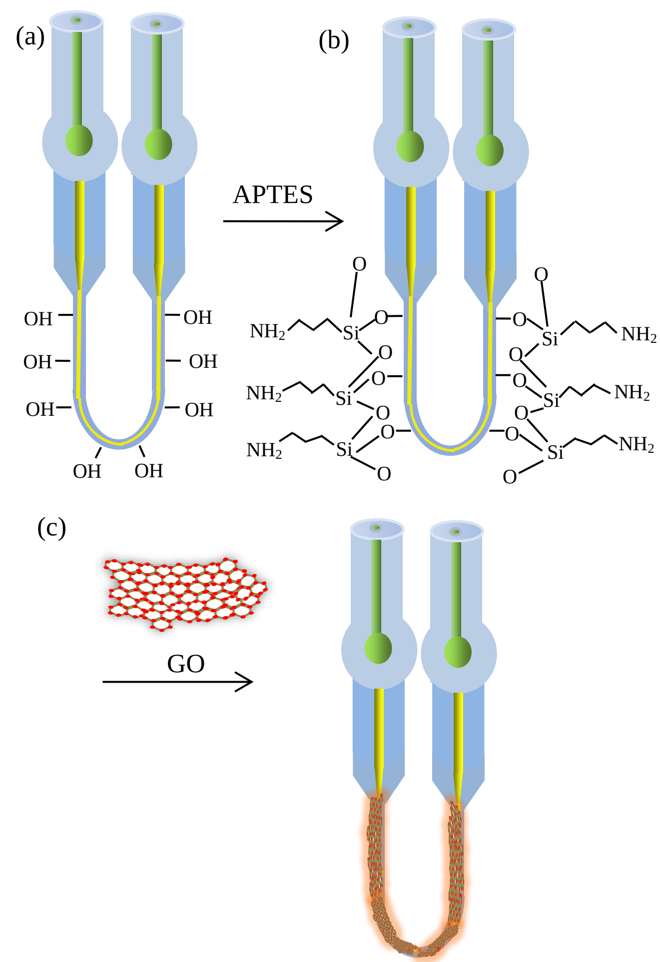



Fig. 4. Specific steps of modifying GO film on the surface of U-shape sensor: (a) producing abundant silanol groups; (b) forming the Si-O-Si chemical bond by APTES; (c) depositing GO on the surface of U-shape microfiber sensor

The SEM images of the microfiber sensor coated with GO film are presented in Fig. 5, indicating that the GO film was coated on the fiber surface tightly and smoothly.

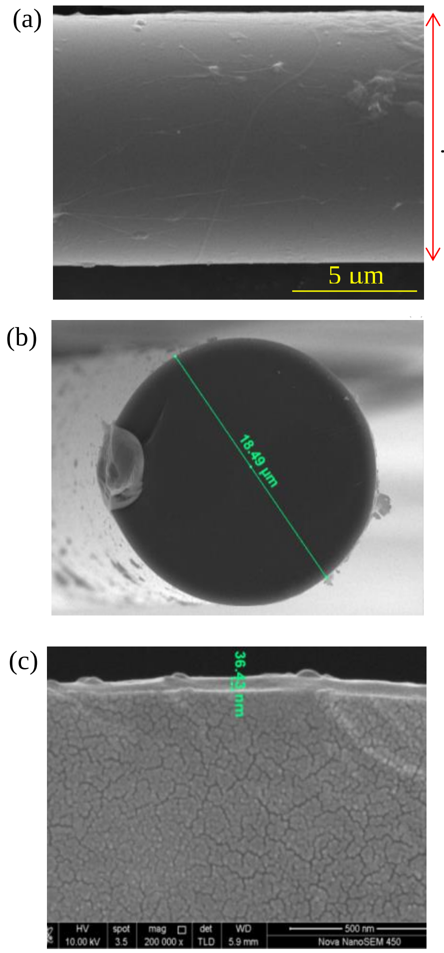



Fig. 5. SEM images: (a) GO coated microfiber surface (b) cross-section of U-shape microfiber sensor modified with GO at a concentration of 0.01 mg/mL; (c) GO film thickness with a concentration of 0.01 mg/mL

## III. Experimental Results and Discussion

Figure 6 shows a schematic diagram of the experimental setup. The U-shape microfiber RH sensor is placed in a RH chamber (ST-80L Xiamen Yishite Instruments Co. Ltd). The light emitted from the broadband light source (BBS, SC-5-FC) transmits through the U-shape optical fiber sensor and output light was monitored by an optical spectrum analyzer (OSA, YOKOGAWA AQ6370D). During the experiment, the temperature in the RH chamber was fixed at a constant temperature of 25 °C and the RH was changed gradually from 30% to 98%RH.

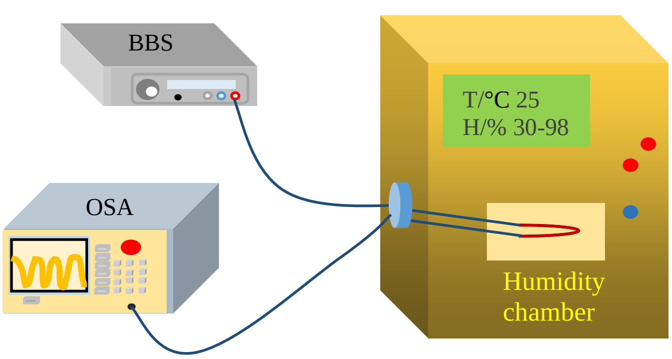
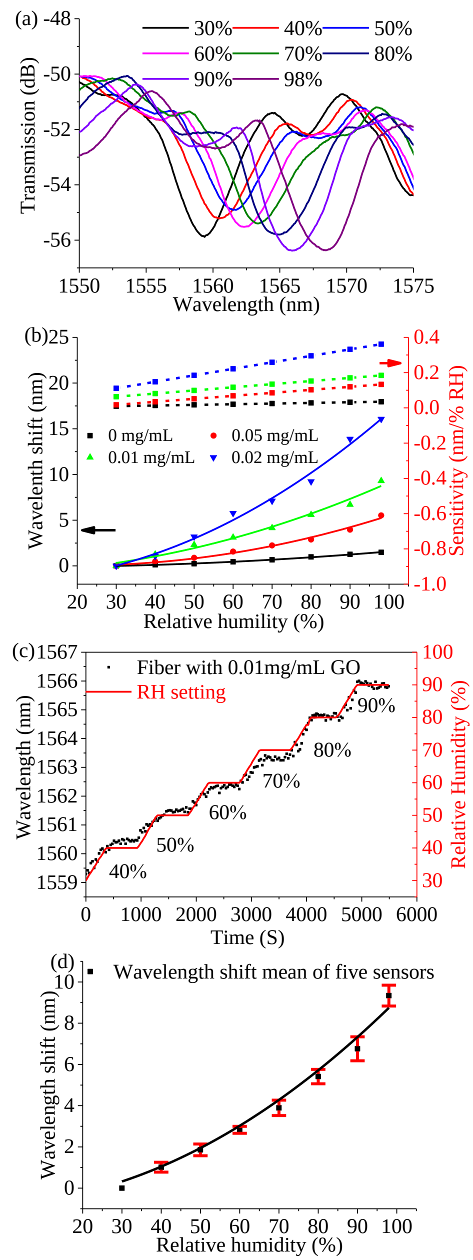



Fig. 6. A schematic diagram of experimental setup

Figure 7 (a) shows the spectral response of the functionalized U-shape microfiber RH sensor coated with 0.01 mg/mL GO solution. As the RH increases from 30% to 98%, the dip wavelength shifts to the longer wavelength monotonically. Figure 7(b) illustrated the dip wavelength shifts of the microfiber sensor functionalized with different concentration of GO solutions (0.01, 0.02 and 0.05 mg/mL) vs. different RH. For the U-shape microfiber sensor without GO film. Second order polynomial fits were applied to these measurements and the sensitivities were plotted by calculating first order derivatives to the fitted functions as shown in Fig. 7(b). The RH sensitivity varies from 0.010 to 0.035 nm/%RH in the RH range from 30% to 98%RH; the RH sensitivities of the three GO coated U-shape microfiber sensors with GO solution of 0.01, 0.02 and 0.05 mg/mL are in the range of 0.063 - 0.184 nm/%RH, 0.111 - 0.361 nm/%RH and 0.017 - 0.133 nm/%RH, respectively in the RH range of 30%-98%. During the experiments, the RH increases gradually from 30% to 40% RH and then stabilize for 20 minutes. This process is repeated until the RH reached to 98%RH.




(a)

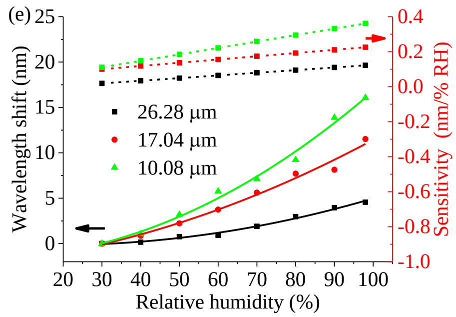



Fig. 7. (a) Spectral response of U-shape microfiber sensor modified with 0.01 mg/mL GO solution; (b) measured wavelength shift and calculated sensitivity vs. RH for the four different U-shape RH sensor; (c) wavelength shift vs. the time changes; (d) reproducibility test of the U-shape microfiber sensor; (e) the wavelength shift and sensitivity of U-shape microfiber sensors with three different taper waist diameters.

Figure 7(c) summarized the wavelength shifts over time for the U-shape microfiber RH sensor functionalized with GO (0.01 mg/mL). The measured wavelength shift of the fiber sensor agrees well with the setting RH values. In order to study the reproducibility of the microfiber RH sensor, 5 U-shape microfiber sensors were fabricated with the same fabrication parameters and modified with the same GO solution of 0.01 mg/mL. The measured wavelength shifts of the 5 sensors at different RH value were shown in Fig. 7(d), where an average RH sensitivity from 0.045 to 0.207 nm/%RH has been achieved. The influence of taper waist diameter of RH sensor has been studied by coating the same thickness GO film (with 0.02 mg/mL GO solution) on the surface of the U-shape microfiber structure. Three different taper waist diameters of 26.28 μm, 17.04 μm and 10.08 μm have been studied and the RH sensitivities are 0.019 - 0.122 nm/%RH, 0.1 - 0.225 nm/%RH and 0.111 - 0.361 nm/%RH respectively as shown in Fig. 7(e), using the same method in Fig. 7(b). It shows that the smaller the diameter, the higher the RH sensitivity provided same thickness of GO film was coated on the surface of the fiber sensor.

Response time is an important indicator for judging the performance of RH sensor. To accurately measure the response time of the microfiber RH sensor, a photodetector (THORLABS: PDA10CS-EC 700-1800 nm; InGaAs-APD) is used to monitor the power variations of the fiber sensor as shown in Fig. 8(a), where the optical signal was detected by a photodetector and processed by a data acquisition card (DAQ; National Instruments NI USB-6000 series). To generate instant RH, a volunteer gently blows to the fiber sensor so that RH changes immediately taken place on the sensor when the volunteer starts and stop to blow. The signal response of RH sensor is shown in Fig. 8(b) as the volunteer starts to blow (RH increases) and stop to blow (RH decreases). Figure 9(c) show that the response time and recovery time of the sensor calculated by the time difference between the 90% signal maximum and the 10% signal maximum points are 0.28 and 0.15 s respectively, which is faster than that of the previous reports in references [34-35].

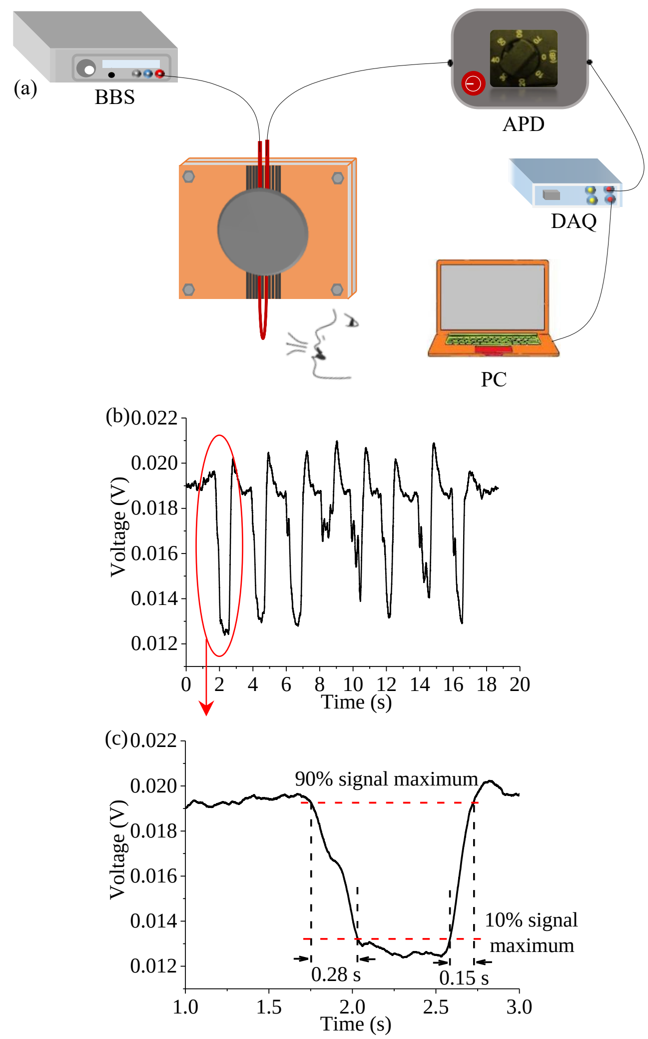
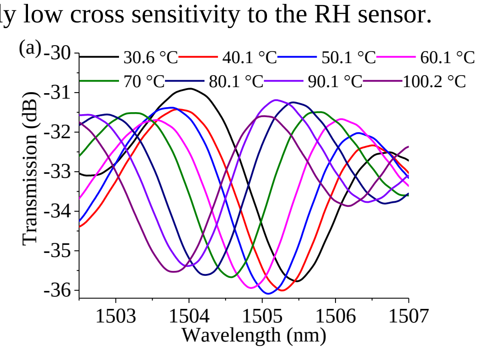



Fig. 8. (a) Schematic diagram of experimental device for detecting response time; (b) response to human breathing of U-shape sensor; (c) response time of sensor

Final, the influence of temperature cross response of the sensor on the RH sensor (with and without GO film) was experimentally studied, and the measured spectral response and wavelength shifts were shown in Fig. 9. Figure 9(a) shows the spectral response of the U-shape sensor without GO film has a blue shift as the temperature increases. Figure 9(b) shows that the average temperature sensitivities of the sensor with and without GO film are -0.02676 and -0.02413 nm/°C, respectively, indicating a good temperature repeatability and relatively low cross sensitivity to the RH sensor.



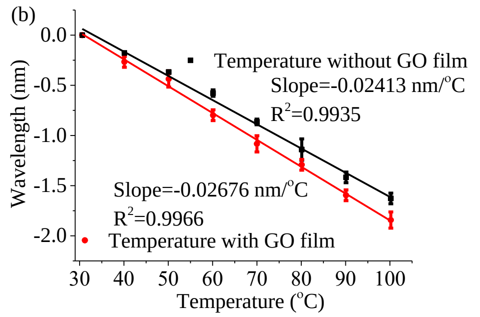



Fig. 9. (a) Spectral response of U-shape optical fiber sensor with a diameter of 10.08 μm without GO film in the temperature range of 30.6 °C-100.2 °C; (b) Temperature sensitivity of sensors without GO film and with GO film.

Table 1 summarized the RH detection range, sensitivity and response time of optical fiber RH sensors with different structures coated with GO film. The sensitivity of our proposed sensor is better than those depicted in [36, 43] in terms of measuring wavelength shift from the comparison. The response time of our proposed sensor is faster than those reported in [37, 39, 41].

TABLE I
PERFORMANCE OF OPTICAL FIBER RH SENSORS WITH DIFFERENT STRUCTURES COATED WITH GO

| Fiber Structure | RH range | Sensitivity (dB/% RH or nm/%RH) | Response time | Reference |
|---|---|---|---|---|
| TFG | 30%-80% | 0.0185 nm/%RH | 0.042 s | [36] |
| Polymer optical waveguide | 60%-100% | 0.553 dB/% RH | <1 s | [37] |
| TFBG | 10%-80% | 0.129 dB/% RH | NA | [38] |
| Side-polished twin-core fiber | 60%-62.1% | 3.76 dB/%RH | 3.6 s | [39] |
| LPG | 60%-95% | 0.15 dB/% RH | NA | [40] |
| Side-polished fiber | 32%-85%<br>85%-97.6% | 0.145 nm/%RH<br>0.915 nm/%RH | 2.73 s | [41] |
| S fiber taper | 84%- 95% | -0.361 dB/%RH | NA | [42] |
| Microfiber-Knot resonator | 0%-80% | 0.0104 nm/%RH | NA | [43] |
| Micro-nano fiber Bragg grating | 20%- 80% | 0.0174 nm/%RH | 3.2 s | [44] |
| **U- shape microfiber** | **30%-98%** | **0.111-0.361 nm/%RH** | **0.28 s** | **Proposed in this paper** |

## IV. Conclusion

In conclusion, a high sensitivity U-shape tapered PPMF microfiber RH sensor was proposed and experimentally studied in this paper. As the diameter of the taper waist decreases from 26.28 to 10.08 μm, the RI sensitivity increases from 752.2 to 1692.5 nm/RIU. By coating a hydrophilic material GO film on the U-shape microfiber sensor surface, the sensor can achieve high sensitivity to RH. Experimental results show that the maximum RH sensitivity is as high as 0.361 nm/%RH at 98%RH for the U-shape sensor (taper diameter 10.08 μm) coated with 0.02 mg/mL GO solution, which is about 10 times than that of the U-shape microfiber sensor without GO coating (0.035 nm/%RH). In addition, the temperature sensitivity of the U-shape RH sensor is -0.02676 nm/°C. The response / recovery time of the proposed RH sensor is 0.28 / 0.15 s. The RH sensor has advantage of good stability, reproducibility, high RH sensitivity and faster response/recovery time.

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